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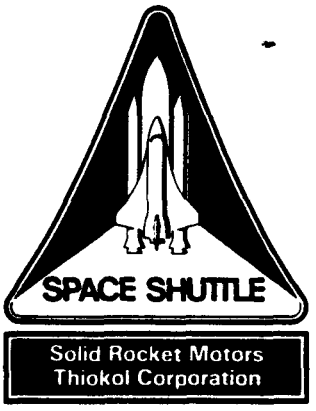

TWR-60140

CHEMLOK[R] 205 PROCESS ANOMALY EVALUATION

FINAL REPORT

FEBRUARY 1990

Prepared for:

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER
MARSHALL SPACE FLIGHT CENTER, ALABAMA 35812

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Thiokol CORPORATION
SPACE OPERATIONS

P.O. Box 707, Brigham City, UT 84302-0707 (801) 863-3511

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PREPARED BY:

D. L. RICE
M&P CASE PREPARATION

APPROVED BY:

Loyal E. Seidner 22 Feb
L. E. SEIDNER
M&P CASE PREPARATION

T. F. Christensen 2/22/90
T. F. CHRISTENSEN, MANAGER
M&P PRODUCTION SUPPORT

D. Marsh 2-20-90
D. M. MARSH
PROGRAM MANAGER

Larry D Allred /for
B. R. MCQUIVEY
SYSTEMS INTEGRATION

P. C. PETTY, MANAGER
CASE PREP ENGINEERING

Thiokol CORPORATION
SPACE OPERATIONS
P.O. Box 707, Brigham City, UT 84302-0707 (801) 863-3511

P C Tydeck 3-2-90
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ECS NO. SS-3627

CONTENTS

FIGURES

TABLE

References: Work Request No. PE90021
LWR No. 583942

Process Engineering Technical Report Categories
Chemlok

1.0 INTRODUCTION AND SUMMARY

After several years of enjoying successful spray applications of Chemlok^{R} 205, the manufacturing work center was suddenly getting process anomalies that were questionable. These anomalies (including dry overspray, and coating thickness in excess of the 0.4 mil maximum allowable) resulted in several PDs being written.

An investigation determined that the primary factors contributing to the process anomalies were excessive air turbulence in the spray pit, high temperatures, and old Chemlok 205. An airflow adjustment in the makeup air system, spraying when the temperature was cooler, and using fresher Chemlok 205 resulted in eliminating the anomalies and all subsequent PDs to date.

2.0 TEST OBJECTIVE

The test objective was to determine the causes of the process anomalies and to make any adjustments necessary to eliminate the questionable anomalies in the Chemlok 205 spray application process.

3.0 CONCLUSION

It was concluded that excessive air turbulence, high temperature in the spray pit, and old Chemlok 205 were the major contributors to process anomalies experienced during the questionable Chemlok 205 applications.

4.0 RECOMMENDATIONS

1. Adjust the make-up air system to prevent forced air from being blown directly into the case segment during the spray application process.

2. Provide temperature control in the make-up air system that will allow capacity for cooling to as low as 75°F for the Chemlok 205, and as high as 135°F for the OD paint curing process.

3. Reduce the shelf life requirement of Chemlok 205 from 12 months maximum to six months maximum.

5.0 DISCUSSION

After Process Departures 164868, 164975, 164992, and 165113 had been written because of excessive Chemlok 205 thickness measurements, it was determined that the spray process was out of control. Representatives from Process Engineering, Case Preparation Manufacturing Engineering, and Case Preparation Engineering Work Center were asked to correct the problem. Process monitoring was immediately initiated to observe all spray applications and identify probable causes for the overthick and overspray conditions.

It was learned during the investigation that the air mover system had been recently modified to provide an increased airflow through the case segment during the spraying operation. The increased airflow from the make-up air system was actually causing air velocities of approximately 800 feet per minute within the case segment. This excessive turbulence was deflecting the spray pattern enough to increase the distance traveled from the spray tip to the case wall, resulting in the Chemlok being too dry to flow evenly after reaching the case wall. The turbulence (caused by forced air being introduced into the top of the segment faster than the air movers capacity to remove it at the bottom of the segment) also contributed to the overspray condition known as cobwebbing.

The three recommendations previously listed have been or are being addressed. The recommended adjustment to the make-up air system was accomplished by changing the air direction louvers; thus, preventing the airflow from being forced directly into the case segment. The temperature control system is being worked on FEO No. 4BV07 by Facilities Engineering to provide environmental control for the make-up air. An Engineering Change Request (ECR 2905) has been submitted, to request a change in the shelf life requirement that will prevent the use of old Chemlok 205.

After the airflow was adjusted to redirect the flow of make-up air, the Chemlok remained wet on the case wall for at least one full revolution of the case. This improvement alone allowed the Chemlok to reach the case wall before excessive flashing of the diluent, resulting in a consistent flow of the material over the case wall while the Chemlok was still liquid. Visual evidence of this improvement can be seen in the 24X photographs (Figures 1 and 2).

PHOTO NO. 1 (Figure 1)

Photo No. 1 is a magnified photo of an ASTM test panel with the pre-spray test coating of Chemlok 205 on 28 August 1989. The Lot 87 (Vendor Lot GJB) material was 10 months old. The average thickness measured on the test panel was 0.72 mils. Since the material did not meet the thickness requirement, a newer lot (No. 111, manufactured April 1989) was issued. A satisfactory spray application within the 0.4 mil maximum thickness requirement was accomplished using the newer material.

An important observation was made during the visual comparison of the two materials. It was noted that the older Chemlok 205 had approximately twice the amount of solids precipitated from suspension (in the paint can) as was observed in the newer lot of Chemlok 205. The excessive amount of solids in the older material made mixing more difficult. However, overspray and cobwebbing were still observed after the Lot 111 application.

PHOTO NO. 2 (Figure 1)

Photo No. 2 is of the ASTM test panel (with its pre-spray test coating) prepared prior to the Chemlok 205 spray application on 9-11-89. The Lot 103 (Vendor Lot GIH) material was 12 months old. The average thickness measured on the test panel was 0.50 mils. The Lot 103 material was discarded and a successful spray application was accomplished using Lot No. 111 material manufactured in April 1989. Overspray and cobwebbing was observed after application of the Lot 111 material.

PHOTO NO. 3 (Figure 2)

Photo No. 3 is of the ASTM test panel (with its pre-spray test coating) prepared prior to the Chemlok 205 application on 11 September 1989, using Chemlok from Lot 111. The average coating thickness on this test panel was acceptable, measuring 0.34 mils. Overspray and cobwebbing was still a problem.

PHOTO NO. 4 (Figure 2)

Photo No. 4 is of the ASTM test panel (with its pre-spray coating) prepared prior to the Chemlok 205 application on 9-23-89, using material from Lot 99. The Lot 99 Chemlok 205 was six months old (manufactured in March 1989). Adjustment of the make-up air system was completed prior to this application. No overspray or cobwebbing was visually detectable. Material flowed evenly and resulted in a coverage notably superior to previous applications.

It is evident that the thickness measurement does not necessarily relate directly to the actual amount of material on the sprayed surface (Table I). The Accuderm coating thickness measurement system uses a magnetic field reading to determine the distance between the probe and the ASTM test panel. When the surface of the measured coating is uneven, the probe will be reading the thickness of the high points rather than a realistic thickness average.

The smoother application of Chemlok 205 is superior, because the smooth surface indicates that the material has been applied in its liquid state, allowing it to flow evenly over the sprayed surface. This will result in increased capacity for mechanical bonding at the primer-to-steel bond interface because the material will have flowed sufficiently to fill all the depressions and surround all the peaks of the properly prepared bond surface.

The chemical bond at the primer (Chemlok 205) to adhesive (Chemlok 233) bond interface is not dependent on surface roughness. This bond is only interested in full coverage contact of the two materials.

The following information has been taken from Lord Corporations Chemlok 205/Chemlok 220 Application Manual (LL-322), by E. L. Polaski.

Preparation Of Adhesives

For single gallon and five gallon pails, adequate mixing is usually achieved by hand stirring in a figure 8 motion, from the bottom, with a wood paint stick. The material should be agitated until all settling is removed from the bottom and stirred back into solution. Frequent stirring should then be employed during the use of the material. Lids should be kept in place when the container is not being used. Failure to do so will result in loss of solvent, reduced solubility of the ingredients, increased solids content and increased viscosity.

Time Of Agitation

It is impossible to specify the exact amount of agitation required to achieve thorough mixing under varying conditions. Agitation should be continued until the solids are completely dispersed and the liquid is homogeneous. For gallon containers, fifteen minutes on a paint shaker is sufficient if the solids have first been broken loose from the bottom.

Chemlok adhesives may be agitated or pumped continuously over indefinite periods without damage. There is no danger of flocculation or precipitation due to over-mixing. The only adverse effect from over-mixing is the loss of solvent occasioned by temperature rise.

Spray Application

The spray method of applying adhesives is particularly applicable in instances where it is desirable to coat only one side or only certain areas of a part. When spraying, it is necessary to have the adhesive reach the metal part wet. If drying occurs in the air before reaching the metal, poor adhesion will result. Excessive cross-ventilation in the spray booth usually aggravates cobwebbing.

Control Of Chemlok 205

Viscosity is not a good control method for Chemlok 205 if a high degree of dilution is employed. The system rapidly reaches the viscosity of almost pure solvent where changes in concentration have very little effect upon the viscosity. Therefore, if it is necessary to work with a dilute cement, control must be achieved by gravimetric measurement of the total solids content. Total solids may be determined by measurement of the residue after forced air drying a thin film for 60 minutes at about 200°F, and should rarely drop below 19 percent.

6.0 INSTRUMENTATION

6.1 Viscosity

A Zahn viscometer (No. 2 cup) was used to measure viscosity.

6.2 Gel Permeation Chromatography (GPC) Analysis

The HP 1090 Chromatograph having the capacity of molecular measurement within the range of 100 Å to 10,000 Å was used for the GPC analysis. It is located in Building 53, Room 7.

6.3 Particle Size Analysis

The Leeds and Northrup Model 7991 Microtrac with a small particle attachment, located in Building 53, Room 12C was used to perform the particle size distribution analysis.

7.0 PHOTOGRAPHY

Magnified (24X) photos of Chemlok 205 coatings on ASTM test panels were made using photomicrograph equipment in the R&D Lab. The photos were helpful in determining why there was no apparent correlation between the coating thickness measurement and the weight of the material.

8.0 TEST DATA REQUIREMENTS

The data are required to support any process changes and material storage life changes necessary to eliminate further process anomalies in the Chemlok spray application process.

9.0 TEST IMPLEMENTATION AND RESULTS

9.1 Viscosity

A Number 2 Zahn cup was used to compare viscosities of aged Chemlok 205 (Lot 109, manufactured October 1988) with viscosities of newer Chemlok 205 (Lot 128, manufactured September 1989) after various mixing and rest periods.

The mixing was accomplished by a lab technician on the paint shaker in Building M-111.

Prior to the first mixing cycle, the precipitated "solids" were measured in each can. The older Chemlok (Lot 109) had a nominal three inches of firmly packed precipitated solids in the bottom of the can compared with a nominal ½-in. of loosely packed precipitated solids in the newer Chemlok (Lot 128).

The steps and resulting viscosity measurements in the mix/rest versus viscosity evaluation are as follows:

(The viscosities reported are each an average of five measurements.)

Step		Average Viscosity (Seconds) Lot 109	Lot 128
1.	Viscosity of Unmixed Chemlok	34.0	35.0
2.	Mixed 5 Minutes On Paint Shaker	32.6	27.8
3.	Mixed 10 Minutes On Paint Shaker	32.4	27.0
4.	Mixed 15 Minutes On Paint Shaker	32.0	27.0
5.	After 5 Minute "Rest" Period	32.0	27.0
6.	After 15 Minute "Rest" Period	34.2	28.4
7.	After 30 Minute "Rest" Period	34.4	29.0
8.	Additional 10 Minutes On Paint Shaker	31.0	26.6

A lower viscosity was recorded after each of the mix and rest periods in the newer material, with less total variation in viscosity measurements after the initial five-minute mix cycle.

It is concluded from this evaluation that the fresher materials will have a better capacity to maintain a consistent viscosity after they are mixed.

9.2 Gel Permeation Chromatography (GPC) Analysis

The GPC analysis, as reported in LWR No. 583942, indicates that no polymerization of the material occurs because of age.

9.3 Particle size Analysis

The particle size analysis, as reported in LWR No. 583942, indicates that there is as much variation within samples from the same lot of material as there is in samples from the various lots of material.

9.4 General Discussion

Mr. E. L. Polaski (Lord Corporation) was contacted for the purpose of discussing the results of our testing. Mr. Polaski stated that there was no particular reason why the viscosities of the unmixed liquids were nearly the same. He did say, however, that the test was not considered of any consequence since the materials should be mixed before checking viscosities. The higher viscosity seen in the older material was not a concern. Both cans of material measured within the normal viscosity range of 20 seconds to 40 seconds expected with a Zahn No. 2 cup.

When Chemlok 205 goes out of spec for viscosity, it will be because of low viscosity (less than 20 seconds with the Zahn No. 2 cup).

Mr. Polaski supports the findings in the GPC analysis because of the ages of the materials tested. Polymerization would not be expected to be detected in the material until it was much older than one year.

The solids found in each of the cans was no surprise. The Chemlok 205 contains 22 to 26 percent solids per manufacturing specification and the solids can be expected to precipitate from suspension "rather rapidly".

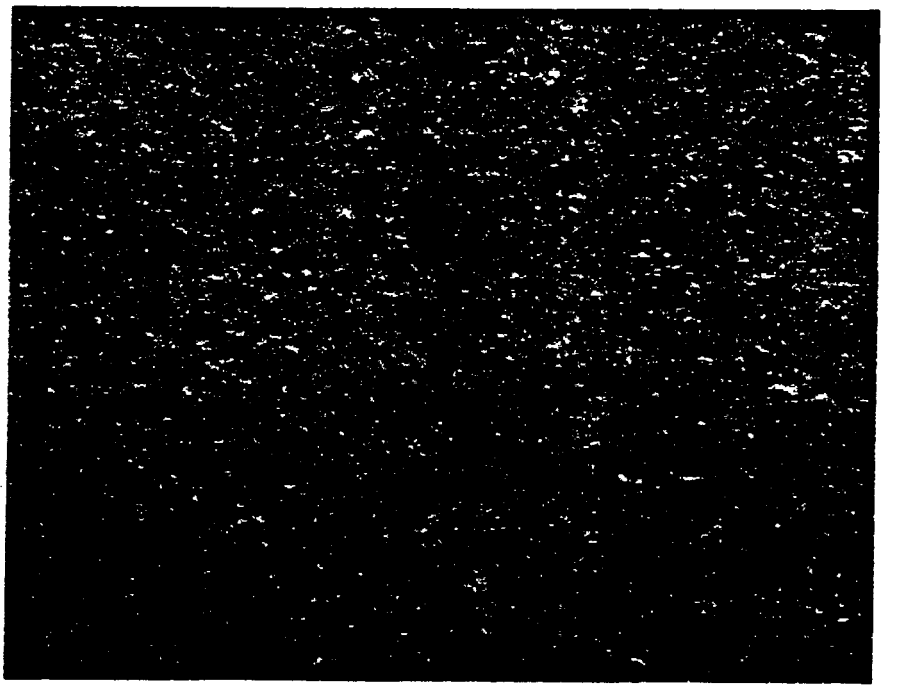

Photo No. 2 (Test Panel No. 5)

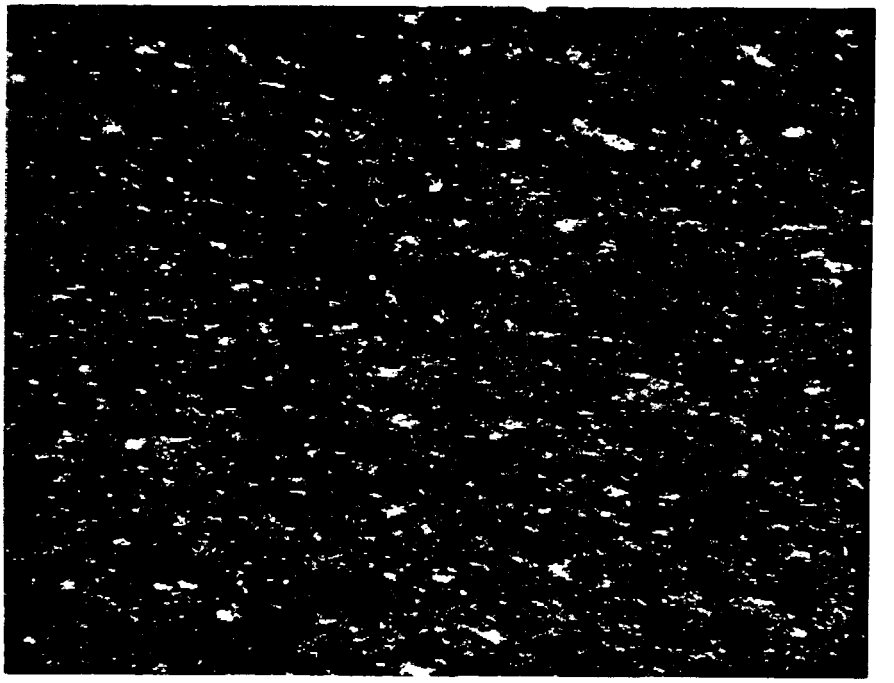

Photo No. 1 (Test Panel No. 1)

Figure 1. Magnified Photos (24X) of ASTM Test Panels (Nos. 1 and 5)

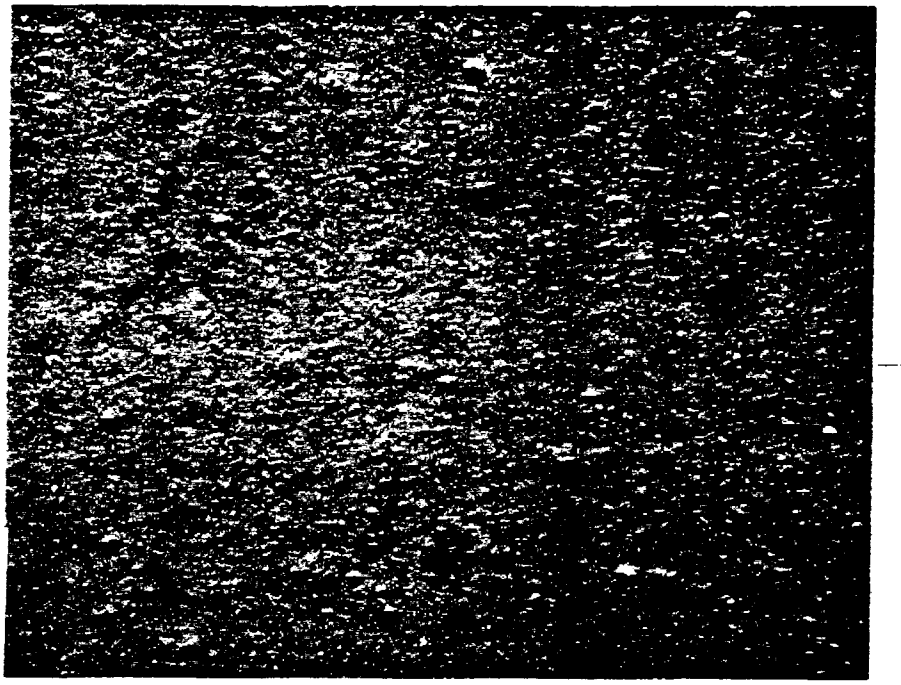

Photo No. 4 (Test Panel No. 22)

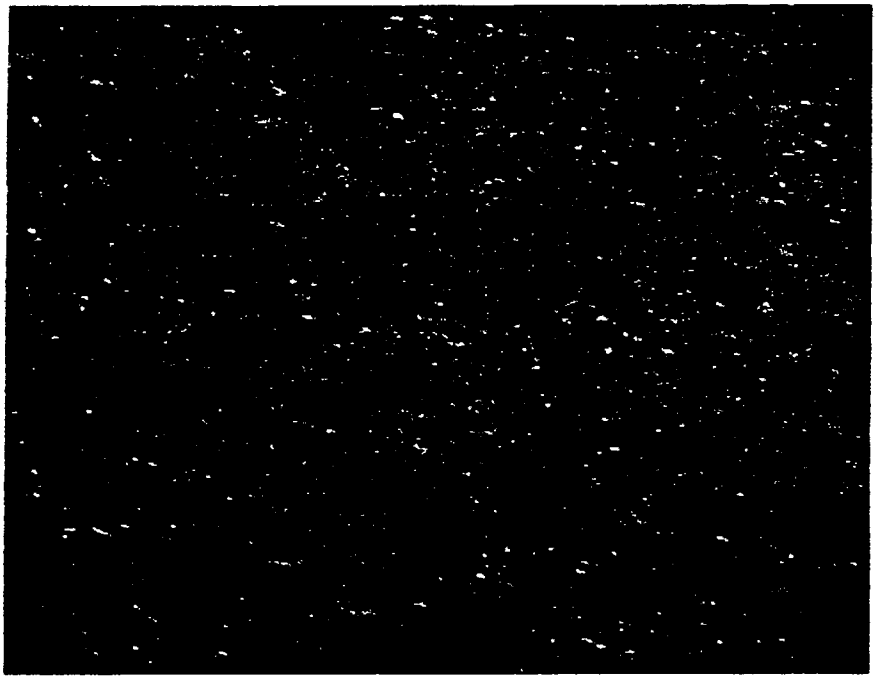

Photo No. 3 (Test Panel No. 12)

Figure 2. Magnified Photos (24X) of ASTM Test Panels (Nos. 12 and 22)

TABLE I. Coating Thickness Versus Coating Weight

Test Panel	Accuderm Thickness Average (mils)	Weight of Panel w/205 (grams)	Weight of Panel Cleaned (grams)	Weight of 205 (grams)	Surface Area Covered (sq in)	"205" per sq. in. (mg)	Lot
1	0.72	131.2949	130.9297	0.3652	37.2	9.82	87
2	0.36	130.0545	129.7323	0.3222	40.5	7.96	103
3	0.38	130.7935	130.4512	0.3423	40.6	8.43	103
4	0.27	131.3830	131.0911	0.2919	39.6	7.37	103
5	0.50	126.6451	126.3153	0.3298	40.9	8.06	103
6	0.41	127.5278	127.1765	0.3513	40.5	8.67	103
7	0.38	127.1232	126.7909	0.3323	41.7	7.97	103
8	0.39	127.1265	126.7908	0.3357	42.1	7.97	103
9	0.32	127.3606	127.0823	0.2783	42.2	6.59	103
10	0.32	126.9682	126.6957	0.2725	41.1	6.63	103
11	0.35	127.1565	126.8368	0.3197	37.9	8.44	103
12	0.34	128.6970	128.4027	0.2943	38.6	7.62	103
13	0.35	128.0719	127.6939	0.3780	42.3	8.94	101
14	0.31	129.0958	128.7643	0.3315	42.6	7.78	101
15	0.34	127.4783	127.0999	0.3784	42.9	8.82	101
16	0.35	127.0626	126.7026	0.3600	42.9	8.39	101
17	0.36	126.4856	126.1977	0.2879	37.0	7.78	101
18	0.37	124.0843	123.8031	0.2812	37.1	7.58	101
19	0.40	125.2867	124.9019	0.3848	41.5	9.27	101
20	0.40	124.8983	124.5119	0.3864	40.6	9.52	101
21	0.35	128.0909	127.7364	0.3545	40.0	8.86	99
22	0.34	127.5370	127.1899	0.3471	40.9	8.49	99
23	0.33	125.1286	124.7700	0.3586	37.5	9.56	99
24	0.34	124.5306	124.1742	0.3564	37.6	9.48	99